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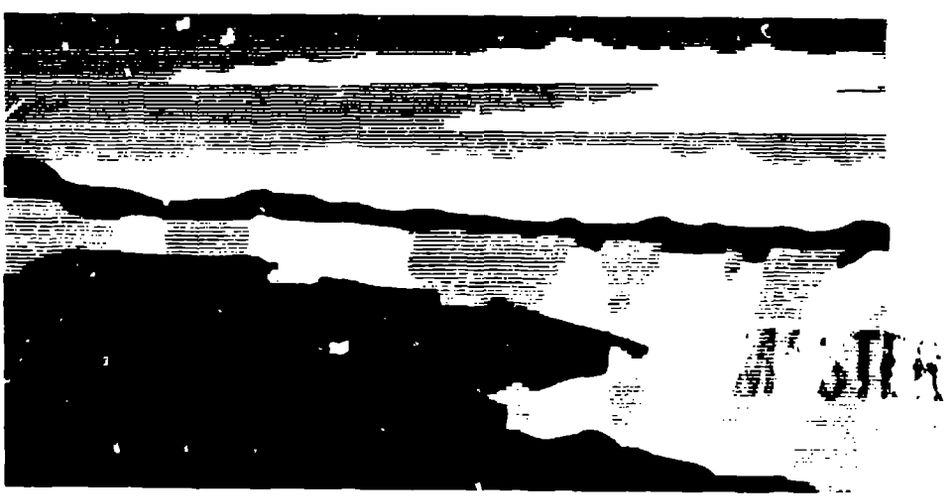

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EFFECT OF ANNEALING TEMPERATURE ON THE TEXTURE OF ROLLED TANTALUM AND TANTALUM-10 WT.% TUNGSTEN

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ABSTRACT

The effect of annealing temperature on crystallographic texture was systematically investigated on straight- and cross-rolled Ta and cross-rolled Ta-10wt.%W sheets. Textures were measured on coupons cut from the rolled sheets and annealed for one hour at temperatures ranging from 800 to 1550°C. In particular the effect of annealing temperature on the volume fraction of grains having (111) and (100) crystal directions nearly aligned with the plate normal was investigated. In the pure tantalum sheets, increased annealing temperature lead to increased fractions of the (111) component. The same was true for the Ta-10wt.%W but much higher temperatures were required to change the texture from a (100) type texture to a more (111) type texture. In addition, the influence of the annealing temperature on the sharpness of the texture; on the surface to centerline texture gradient; and on the in-plane symmetry of the texture was investigated.

INTRODUCTION

The properties of tantalum, like all bcc materials, are strongly influenced by crystallographic texture. Thus, a clear understanding of the development of texture during the processing of polycrystalline tantalum is of considerable value. A good understanding of the processing/texture relationship enables more optimal processing routes to be designed for tailoring the material to have a desired set of properties. Clark and coworkers [1, 2, 3] have investigated the effects of different processing variables on the texture in tantalum. They focused on the influence of different deformation steps on the resulting as-processed and final recrystallization textures. This work explores, in detail, the effect of annealing temperature on the texture of straight-rolled and cross-rolled tantalum sheet as well as on cross-rolled tantalum-10wt.% tungsten sheet.

EXPERIMENTAL DETAILS

The starting plate materials used in this study were commercially pure tantalum and a tantalum alloy containing 10 wt.% tungsten. The chemical composition for both materials is reported in table I. Significant chemical banding was observed in the Ta-10wt.%W indicating that the

temperature was not high enough to fully homogenize the material. Both starting plates were cross-rolled and fully recrystallized.

Table I - Chemical Compositions (in ppm wt. %)

Metal (thickness)	C	O	N	H	W	Nb	Si	Ta
Ta (0.400")	10	<50	<10	<5	<25	<25	<5	bal.
Ta-10W (0.600")	11	63	<10	<5	9.6%	385	5	bal.

The pure tantalum plate was reduced from 0.400" thick plate to 0.030" thick sheet. One of the principle directions in the plane of the starting plate was arbitrarily chosen as the longitudinal direction and the other principle direction was denoted the transverse direction. Three different samples of the starting plate were reduced to 0.030" sheet. One sample was straight-rolled parallel to the longitudinal direction of the starting plate; a second sample was straight-rolled parallel to the transverse direction of the plate and a third sample was cross-rolled. Each of samples were rolled using the same schedule. The plates received uniform though-thickness deformation in each pass. No intermediate anneals were performed.

Fully annealed rolled Ta-10wt.%W was reduced from 0.600" thick plate to 0.030" thick sheet by cross-rolling. However, this material received intermediate anneals in order to prevent break-up.

Coupons were cut from each of the rolled sheets and annealed for one hour hold times for temperatures ranging from 800 to 1550°C in 50° increments. The coupons were annealed under vacuum and quenched in argon.

An inspection of the micrographs from the longitudinal and transverse sections of the rolled tantalum sheets indicated that recrystallization starts as low as 800°C. Different degrees of partial recrystallization are achieved at temperatures between 800°C and 1100°C after which the micrographs suggest that the material is fully recrystallized. In the Ta-10wt.%W the microstructure looked nearly completely recrystallized at 1550°C.

Microhardnesses were measured using a Vicker's indenter on transverse and longitudinal sections of the rolled and annealed sheet coupons per ASTM standard E 384-90. The values reported here are averages of the measurements from the transverse and longitudinal sections. The ratio between the average hardness measured on the transverse section to the average hardness of the longitudinal section for the same coupon ranged from 0.94 to 1.09.

Textures were measured on the as-rolled sheets as well as on the annealed coupons using X-ray diffraction. (110), (200) and (211) pole figures were measured at the centerlines of the samples. In addition, surface textures were measured on the straight-rolled tantalum sheet. The corresponding orientation distributions were determined from the experimental pole figures using *popLA* [4], a preferred orientation analysis software package developed at Los Alamos. No sample symmetry was enforced during the calculations. For each orientation distribution, the texture index (which is a measure of the sharpness of the texture) was also calculated. Sets of representative weighted discrete orientations were generated after the manner of Kocks *et al.* [5] for each of the orientation distributions calculated. Each set of weighted discrete orientations contained 1152 orientations. These weighted orientations were used to estimate the volume fraction of material having a particular (*hkl*) direction aligned within a given number of degrees of the sheet surface normal, $V^f_{(hkl)}(\theta)$. For each set of weighted discrete orientations, $V^f_{(111)}(15°)$ and $V^f_{(100)}(15°)$ were calculated. In addition, an "in-plane texture" index, Q_f, was calculated (see the Appendix). This parameter describes the departure of a given texture from having an ideally axisymmetric texture fiber aligned with the sample normal.

RESULTS

The influence of the annealing temperature on the volume fraction of grains having (111) and (100) crystal axes aligned within 15° of the sheet normal are shown in figures 1 to 4 for the longitudinal straight-rolled Ta, transverse straight rolled Ta, cross-rolled Ta and cross-rolled Ta-10%W.

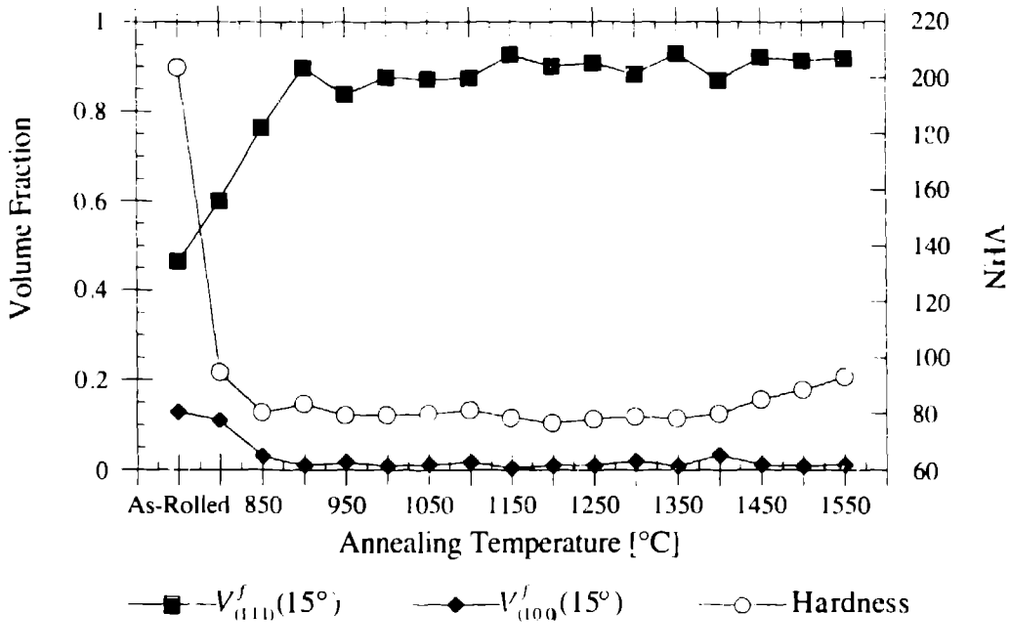

Figure 1. $V^f_{(hkl)}(\theta)$ and hardness versus annealing temperature for longitudinal straight-rolled Ta.

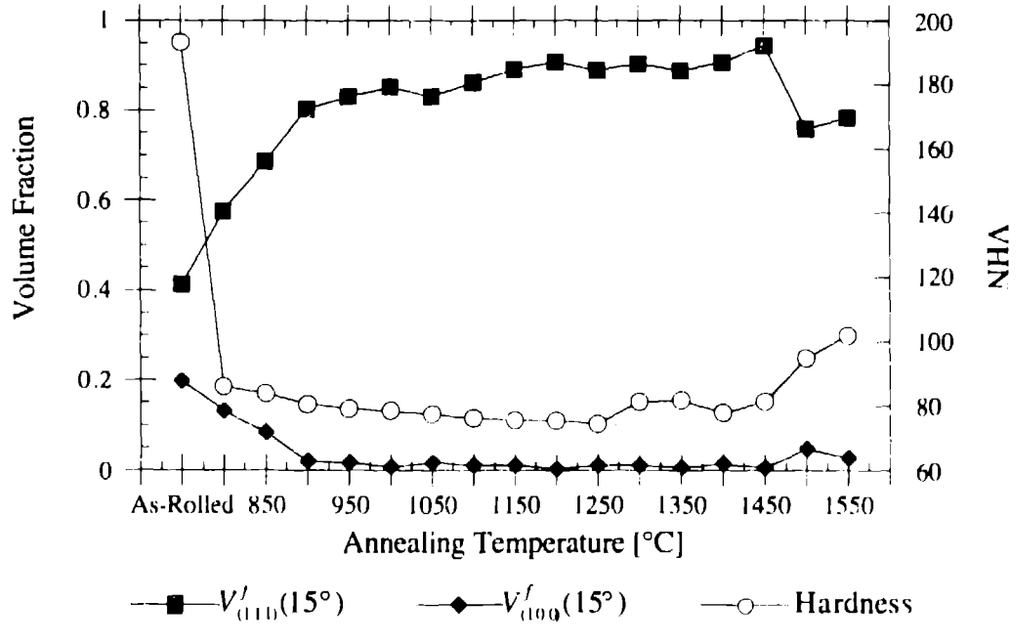

Figure 2. $V^f_{(hkl)}(\theta)$ and hardness versus annealing temperature for transverse straight-rolled Ta.

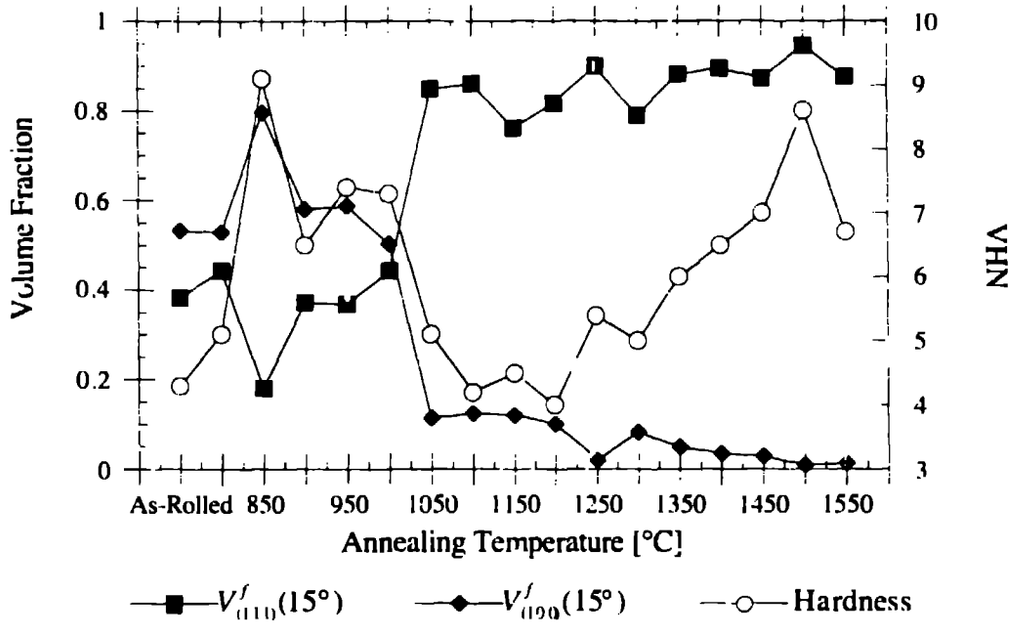

Figure 3. $V^f_{(hkl)}(\theta)$ and hardness versus annealing temperature for cross-rolled Ta.

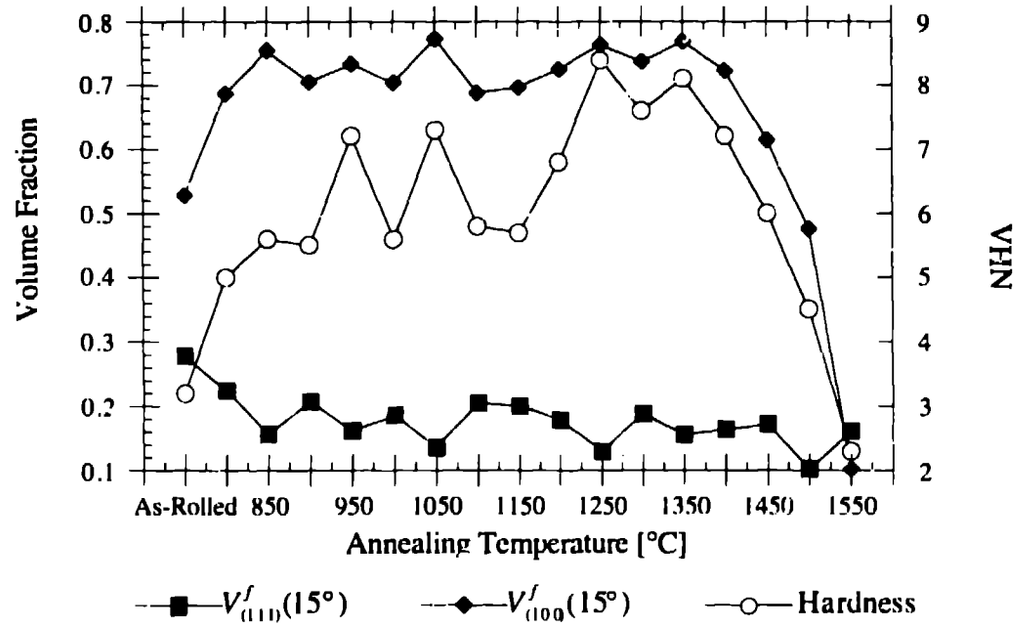

Figure 4. $V^f_{(hkl)}(\theta)$ and hardness versus annealing temperature for cross-rolled Ta-10wt.%W.

The influence of annealing temperature on the through-thickness texture gradient is shown in figure 5 for the longitudinal straight-rolled Ta sheet.

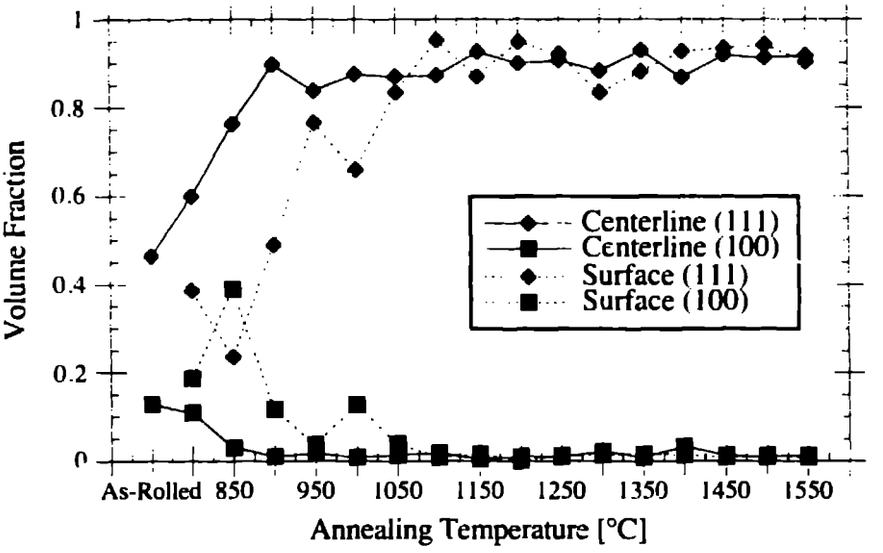

Figure 5. $V^{f}_{(hkl)}(15°)$ versus annealing temperature for longitudinal straight-rolled Ta derived from textures measured at the sheet surface and centerline.

The texture index, which describes the sharpness of strength of the texture, is plotted versus the annealing temperature in figure 6 for each of the rolled sheet materials. A texture index value of one denotes a perfectly random texture.

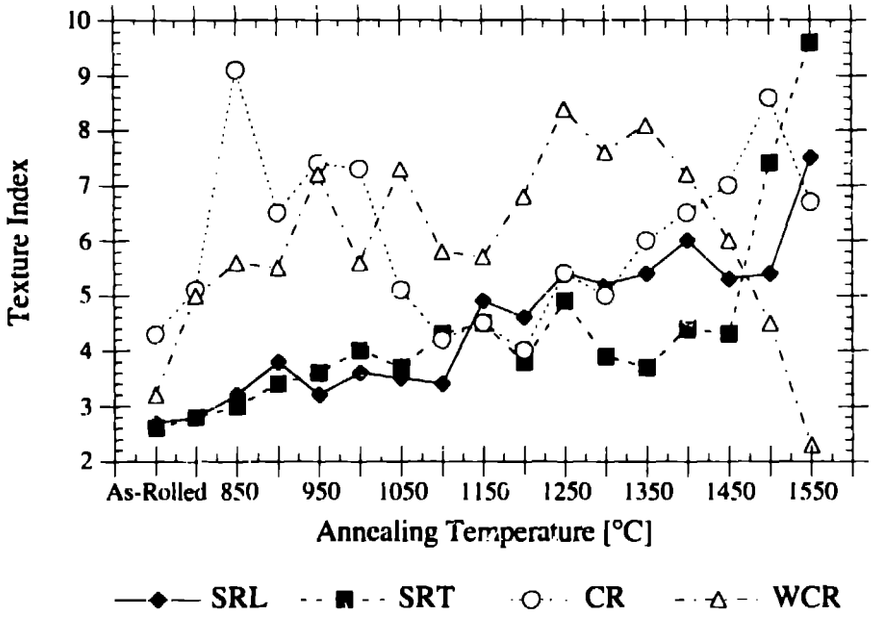

Figure 6. Texture index versus annealing temperature for longitudinal straight-rolled Ta (SRL), transverse straight-rolled Ta (SRT), cross-rolled Ta (CR) and cross-rolled Ta-10wt.%W (WCR).

Figure 7 shows the amount of in-plane anisotropy in the texture at different temperatures for each of the rolled sheet materials. The in-plane texture index, Q_I, is zero for a perfectly isotropic in-

plane texture (i.e. random rotations about a crystal axis oriented normal to the sheet direction) and tends toward a value of one with increased anisotropy

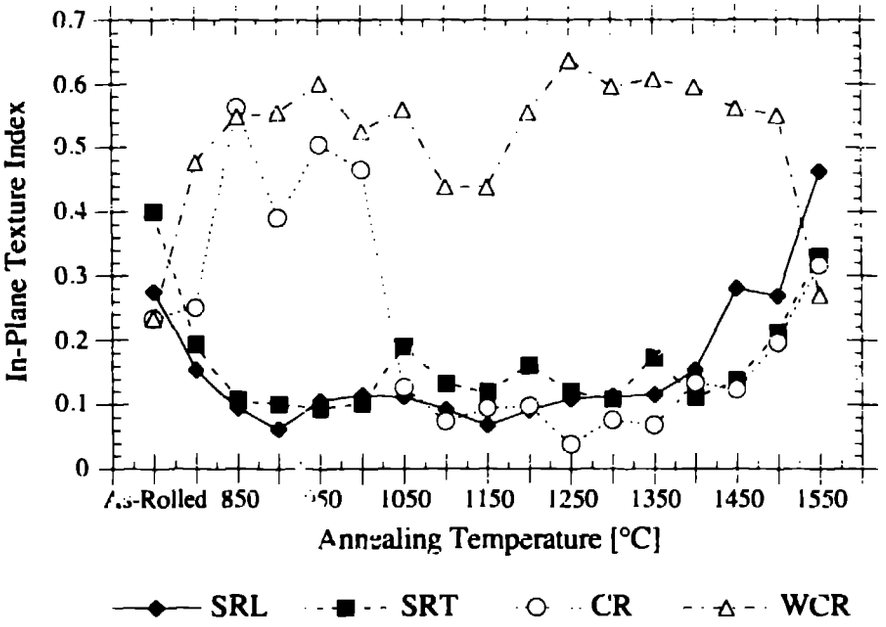

Figure 7. In -plane texture index, Q_{20} versus annealing temperature for longitudinal straight-rolled Ta (SRL), transverse straight-rolled Ta (SRT), cross-rolled Ta (CR) and cross-rolled Ta-10%W (WCR).

DISCUSSION

As can be seen in figures 1-3 for the pure Ta sheets, as the temperature increases the amount of the (111) component (meaning the volume fraction of (111) crystal axes aligned with the sheet surface normal) of the texture increases and plateaus; whereas, the (100) component decreases to zero. The texture evolution of the longitudinal and the transverse straight-rolled material is very similar. The cross-rolled tantalum requires a higher temperature to bring out and stabilize the (111) texture component. The same amount of work was applied to the cross-rolled sample during rolling as the straight-rolled samples. The difference in behavior may be due to the activation of more slip systems in the cross-rolled case. With each change in rolling direction different slip systems will be activated because of the change in boundary conditions.

In the case of the Ta-10wt.%W the (100) texture increases and dominates until about 1350°C; after which, a sharp drop in (100) and a slight increase in (111) occurs. It appears that the texture has begun to transform from a (100) dominated texture to a possible (111) type texture. However, data at higher temperatures is needed to confirm this tendency.

The texture gradient is likely due to a non-uniform distribution in the work applied to the plate during the rolling process at different depths through the thickness of the sheet. Thus certain regions are more resistant to recrystallization than others. Clark *et al.* [1] have described the influence of processing parameters on the strength of texture gradients in rolled tantalum and Wright *et al.* [6, 7] have demonstrated the influence of the gradient on the mechanical response. Figure 4 clearly shows that increased temperature results in a reduction in the strength of the texture gradient. This is probably due to higher temperatures increasing the driving force required for recrystallization events to occur. With increasing driving force, recrystallization is induced in regions in the microstructure resistant to recrystallization at lower temperatures.

The texture increases in strength with increasing temperature for the pure Ta sheets as shown in figure 6. The increase in texture index can be due to two factors: 1) as the temperature increases

not only do more grains have (111) axes nearly aligned with sample normal as shown in figures 1-3 but the (111) axes are also becoming more exactly aligned with the sample normal or 2) as the temperature increases not only do more grains have (111) axes nearly aligned with the sample normal as shown in figures 1-3 but these grains have also become more aligned within the plane of the sheet. The results of the in-plane texture index calculation given in figure 7 show that both the former and latter cases are true. At lower temperatures the in-plane texture decreases; whereas, at higher temperatures the in-plane texture increases. The decrease in in-plane texture at the lower temperatures is accompanied by an increase in the volume fraction of the (111) texture component. This suggests that as the (111) grains align themselves with the sheet normal they tend to be randomly oriented about the plate normal direction. However, at higher temperatures the (111) grains not only become more aligned with the sheet normal but also begin to become more aligned within the plane of the sheet. The data also indicate that when grains have (100) axes aligned with the sample normal they tend to also be aligned within the plane of the sheet regardless of temperature.

The increase in in-plane anisotropy at higher temperatures may explain the rise in the hardness data at higher temperatures in the pure tantalum materials. The grains are becoming oriented in harder orientations with respect to the principal in-plane directions of the sheet. Preliminary Taylor factor calculations seem to bear this out. A Taylor factor of 2.61 was calculated for compression along the transverse axis in the cross rolled tantalum sample annealed at 1200°C and a Taylor factor of 2.96 was calculated for the 1500°C sample.

CONCLUSIONS

In the pure tantalum sheet materials, increasing the annealing temperature yields a greater fraction of (111) type texture. However, higher temperatures are needed to achieve the same effect in the cross-rolled material relative to the straight rolled materials. The cross-rolled Ta-10wt.%W alloy appear to start this trend at about 1400°C, but more data at higher temperatures is needed to confirm the change from a (100) dominated texture to (111) type texture.

Increasing the annealing temperature reduces the strength of the texture gradient. In-plane anisotropy initially decreases with increasing temperature, but gradually increases again at higher temperatures. This is due to an alignment of crystals within the plane of the sheet.

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APPENDIX

An orientation distribution function (ODF) can be represented as a series of generalized spherical harmonics as follows (see Bunge [8] for more detail):

$$f(g) = \sum_{l\mu\nu} C_l^{\mu\nu} T_l^{\mu\nu}(g)$$

where $f(g)$ is the ODF, the $T_l^{\mu\nu}(g)$ terms are generalized spherical harmonics and the $C_l^{\mu\nu}$ are coefficients of the series expansion; g represents a crystallographic orientation. The $C_l^{\mu\nu}$ coefficients can be determined from pole figures or single orientation measurements.

The number of terms in the series expansion can be reduced by enforcing both crystal and sample symmetry. If cubic crystal symmetry and triclinic sample symmetry are assumed then a given set of $C_l^{\mu\nu}$ can be determined. Let them be denoted ${}^{\mathrm{T}}C_l^{\mu\nu}$. If cubic crystal symmetry and cylindrical or axial sample symmetry are assumed then a different set of $C_l^{\mu\nu}$ can be calculated. Let these be denoted ${}^{\mathrm{A}}C_l^{\mu\nu}$. In a sense, the set of ${}^{\mathrm{A}}C_l^{\mu\nu}$ are a subset of the set of ${}^{\mathrm{T}}C_l^{\mu\nu}$. Certain ${}^{\mathrm{T}}C_l^{\mu\nu}$ are forced to zero when axial symmetry is enforced in the calculations. Let the coefficients that are forced to be zero for the case of axial sample symmetry be denoted ${}^{\bar{\mathrm{A}}}C_l^{\mu\nu}$. A measure of in-plane anisotropy can then be defined using the texture index approach set forth by Bunge [8]:

$$Q_l = \frac{\sum_{l\mu\nu} \frac{1}{2l+1} \left| {}^{\bar{\mathrm{A}}}C_l^{\mu\nu} \right|^2}{\sum_{l\mu\nu} \frac{1}{2l+1} \left| {}^{\mathrm{T}}C_l^{\mu\nu} \right|^2}$$

It should be noted that Q_l is bounded: $0 \le Q_l \le 1$. Q_l will be zero when the orientation distribution exhibits perfect in-plane isotropy and will tend toward one as the in-plane anisotropy becomes more marked. In practice, this ratio can be determined by simple manipulation of the ν index of the $C_l^{\mu\nu}$ coefficients determined from pole figures or single orientation measurements.